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(a) ***Cover Page***

Cyclic Imide and Open-Chain Amide Carboxylic Acid Derivatives from the Facile Reaction of *cis*-Cyclohexane-1,2-dicarboxylic Anhydride with Three Substituted Anilines.

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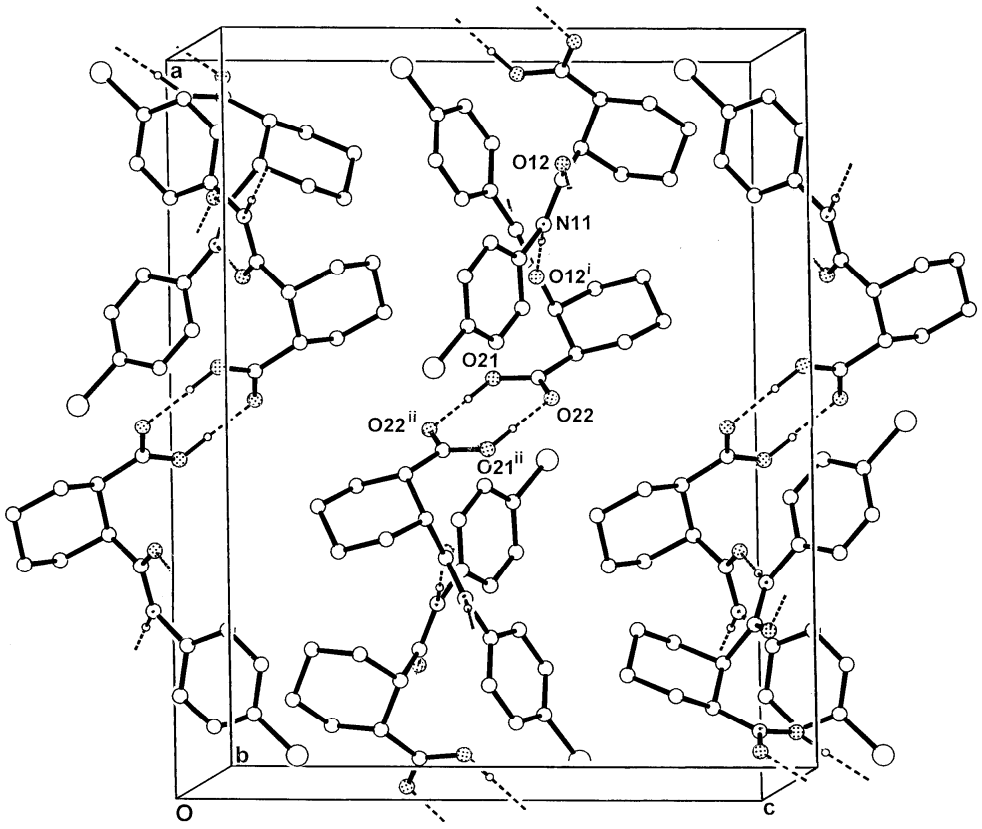
(b) Index Abstract

Graham Smith and Urs D. Wermuth

The structures of an open-chain amide carboxylic acid and two cyclic imides from the facile reaction of *cis*-cyclohexane-1,2-dicarboxylic acid anhydride with the 4-chloro-, 4-bromo- and 3-carboxy-4-hydroxyaniline, respectively, are reported together with their hydrogen-bonding patterns.



(c,d). ***Title: Authors and Affiliations***

Title

Cyclic Imide and Open-Chain Amide Carboxylic Acid Derivatives from the Facile Reaction of *cis*-Cyclohexane-1,2-dicarboxylic Anhydride with Three Substituted Anilines.

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Abstract

The structures of the compounds from the reaction of *cis*-cyclohexane-1,2-dicarboxylic anhydride with 4-chloroaniline [*rac*-2-[(4-chlorophenyl)carbamoyl]-*cis*-cyclohexane-1-carboxylic acid] (**1**), 4-bromoaniline [2-(4-bromophenyl)-3a,4,5,6,7,7a-hexahydroisoindole-1,3-dione] (**2**) and 3-carboxy-4-hydoxyaniline (5-aminosalicylic acid) [2-(3-carboxy-4-hydroxyphenyl)- 3a,4,5,6,7,7a-hexahydroisoindole-1,3-dione] (**3**) have been determined at 200 K. Crystals of the open-chain amide carboxylic acid **1** are orthorhombic, space group *Pbcn*, with unit cell dimensions a = 20.1753(10), b = 8.6267(4), c = 15.9940(9) Å, and Z = 8. Compounds **2** and **3** are cyclic imides, with **2** monoclinic having space group $P2_1$ and Z = 4, with cell dimensions a = 11.5321(3), b = 6.7095(2), c = 17.2040(5) Å, β = 102.527(3)°. Compound **3** is orthorhombic, space group $P2_12_12_1$ with Z = 4 and cell dimensions a =

6.4642(3), *b* = 12.8196(5), *c* = 16.4197(7) Å. Molecules of **1** form hydrogen-bonded cyclic carboxylic acid dimers [graph set $R^2_2(8)$] which are extended into a two-dimensional layered structure through amide-group associations: **3** forms into one-dimensional zigzag chains through carboxylic acid…imide O-atom hydrogen bonds, while compound **2** is essentially unassociated. With both cyclic imides **2** and **3**, disorder is found which involves the presence of partial enantiomeric replacement of the *cis*-1,2-substituted cyclohexane ring systems.

Key Words: *cis*-cyclohexane-1,2-dicarboxylic acid; cyclic imides; open-chain amide carboxylic acids

Running Title:

cis-cyclohexane-1,2-dicarboxylic acid cyclic imides and amide acids

Cyclic Imide and Open-Chain Amide Carboxylic Acid Derivatives from the Facile Reaction of *cis*-Cyclohexane-1,2-dicarboxylic Acid Anhydride with Three Substituted Anilines.

by

Graham Smith* *and Urs D. Wermuth*

Introduction

Cyclohexane-1,2-dicarboxylic anhydride is of interest as a reagent because in its reactions it confers the *cis* configuration [(1*R*,2*S*) or (1*S*,2*R*)] upon the reaction products. With the *cis*-isomer of cyclohexane-1,2-dicarboxylic acid (CHDC), because of the low interconversion potential between these components, unlike the *trans*-isomer [(1*R*,2*R*) or (1*S*,2*S*)], the *dl* enantiomeric pairs are inseparable, forming racemic pairs [1]. The structures of both the racemic and chiral *trans* forms have been determined [2, 3], together with that of the racemic *cis* form [4]. The 1:1 stoichiometric reaction of cyclohexane-1,2-dicarboxylic anhydride with Lewis bases usually gives the racemic hydrogen *cis* -CHDC proton-transfer salts and the structures of a limited number of these have now been determined: the ammonium salt (a dihydrate) [5], the 2-aminopyridinium salt [6], the 4-aminopyridinium salt [7] and the 4-carbamoylpiperidinium salt [8] (all three anhydrous). However, the chiral brucinium salt is also known [9] in which the (1*R*)-carboxylate-(2*S*)-carboxy-*cis*-CHDC species has been captured.

With the substituted anilines, formation of cyclic imides or open-chain amide carboxylic acids may occur, analogous to those formed with phthalic anhydride, (the phthalimides and the phthalanilic acids), often under mild reaction conditions [10]. The structures of the cyclic CHDC imides formed with 5-benzyloxy-2,4-dichloroaniline [11] and with urea [12] were the only ones reported until our recent work [13, 14] provided a number of examples of both cyclic imides and open-chain amide carboxylic acids from the reaction of CHDC anhydride with *X*-monosubstituted anilines (imides: *X* = *o*-F, *p*-F, *p*-OCH_3, *m*-CO_2H, *p*-CO_2H; amide acids: *R* = *m*-F, *o*-OCH_3). All products were obtained under mild 1:1 stoichiometric reaction conditions in aqueous ethanolic solutions.

Herein we report the structures of the crystalline products obtained under similar mild reaction conditions from the 1:1 reaction of *cis*-cyclohexane-1,2-dicarboxylic anhydride with 4-chloroaniline, the open-chain amide carboxylic acid [2-[(4-chlorophenyl)carbamoyl]-*cis*-cyclohexane-1-carboxylic acid] (**1**); with 4-bromoaniline [2-(4-bromophenyl)-3a,4,5,6,7,7a-hexahydroisoindole-1,3-dione] (**2**) and with 3-carboxy-4-hydroxyaniline (5-aminosalicylic acid) [2-(3-carboxy-4-hydroxyphenyl)- 3a,4,5,6,7,7a-hexahydroisoindole-1,3-dione] (**3**) (both cyclic imides).

INSERT 1 Schematic of compounds 1-3 are given here (CHDC3.eps)

The compounds **1**, **2** and **3**

Experimental Section

Preparation.

Compounds **1**-**3** were synthesized by heating together under reflux for 10 min., 1 mmol quantities of *cis*-cyclohexane-1,2-dicarboxylic anhydride and the appropriate aniline (4-chloroaniline for **1**, 4-bromoaniline for **2** and 5-aminosalicylic acid for **3**), in 50 mL of 50% ethanol-water. After concentration to *ca*. 30 mL, partial room temperature evaporation of the hot-filtered solutions gave colourless plates of **1** and **2** or pale brown needles of **3** from which specimens were cleaved for the X-ray analyses.

Crystallography.

X-ray diffraction data for **1**-**3** were acquired at 200(2) K on an Oxford Diffraction Gemini Ultra CCD-detector diffractometer employing graphite crystal monochromatized Mo *K*α radiation (λ = 0.71073 Å). Data collection and reduction and absorption correction (multi-scan) were completed using CrysAlis PRO [15]. The structures were solved using direct methods (SIR92 [16]) and refined with SHELXL97 [17] operating within WinGX [18]. Hydrogen atoms potentially involved in hydrogen-bonding interactions for compounds **1** and **3** were located by difference methods but their positional parameters were constrained in the final refinement cycles with thermal parameters riding [U_{iso}(H) = 1.2 U_{eq}(O)]. Other H atoms in all compounds were included in the refinements at calculated positions and treated as riding with C-H = 0.93-0.98 Å and U_{iso}(H) = 1.2 U_{eq}(C). Disorder was identified within the cyclohexane ring in only the B molecule in **2,** resulting from a partial presence of the [C8(*R*),C9(S)] *cis*-enantiomer (C) [site occupancy factor = 0.27(1)] with the larger occupancy [C8(*S*),C9(*R*)] (B) *cis*-enantiomer (S.O.F = 0.73). The disordered atoms C4C and C7C were subsequently refined isotropically. With **3** the disorder was more extensive, affecting most of the perhydroisoindoline-1,2-dione system, also

from partial replacement of the major [C8(*R*),C9(S)] component [S.O.F = 0.85(1)] by the minor component [C8(*S*),C9(*R*)] (A) [S.O.F = 0.15(1)], which was also refined isotropically. Absolute configuration of the *cis*-CHDC species in compounds **2** and **3** could not be confirmed from the analyses and with **3**, in the absence of a suitable heavy atom in the structure, Friedel pairs (2868) were merged in the final cycles of refinement. General crystallographic details are given in Table 1. The atom numbering schemes employed for all species are shown in Figs. 1-3 [19].

Results and Discussion

In the structure of the racemic open-chain amide carboxylic acid **1** (Fig. 1) the *p*-chlorophenyl ring is rotated slightly out of the plane of the interlinking carboxamide side-chain giving a slight twisting [intra-ring torsion angles C2-C1-C12-N11, C1-C12-N11-C11 and C12-N11-C11-C61: -179.7(2), 173.0(3) and 152.1(3)^{o}, respectively]. Present also is an intramolecular aromatic ring C21-H…O12(ketone) interaction [2.902(3) Å]. The carboxylic acid group is rotated out of the C1-C2-C3 molecular plane of the cyclohexane ring [torsion angle C1-C2-C21-O22, 148.9(2)^{o}]. The 1,2-disubstituted cyclohexane ring as expected has the *cis*-configuration.

INSERT 2 :

Figure 1 (Atom numbering scheme for compound 1) CHDC31.TIF

In the crystal packing the carboxylic acid group forms classic centrosymmetric head-to-head cyclic intermolecular hydrogen-bonding associations [graph set $R^2_2(8)$] [20] [O21-H…O22, 2.662(3) Å: symmetry code (i) -*x* + 1, -*y* + 1, -*z* +

1], the dimers formed being extended into a two-dimensional sheet structure through amide N11-H…O12^{ii}(ketone) hydrogen bonds [2.862(3) Å: symmetry code (ii) $-x + 1, y - ½, z$] (Fig. 2). This structure is similar to that of the analogous compound *N*-(4-chlorophenyl)phthalanilic acid [21].

INSERT 3

Figure 2 (The hydrogen-bonding interactions in the unit cell of (1)

CHDC32.TIF

The two *cis*-cyclic imides **2** and **3** show many structural similarities.

Both compounds are conformationally similar with variations resulting from ring rotations about the imine N-C(phenyl) bond, as indicated by the minimum C-N2-C-C torsion angles of 49.8(4)^o and -61.1(4)^o for the two independent molecules in **2** and -65.7(3)^o in **3**. A common feature of both **1** and **2** is the conformational stress within the cyclohexane ring system as indicated by the relatively higher thermal activities observed in the constituent carbon atoms, resulting in disorder which is more extreme in **3**. This activity has also been present in other examples of cyclic imides of this series [13, 14] but not to the extent observed in **2** and **3**.

With compound **2**, there are two independent cyclic imide molecules (A and B) in the asymmetric unit (Figs. 3, 4). In both of these, the *cis*-configuration is present as expected, with ordered A having a [C8A(*R)*,C9A(*S*)] assignment and the major component of the B molecule, [C8B(*S)*,C9B(*R*)], with the minor (C) component [C8C(*R)*,C9C(*S*)], A and B representing a partial racemic pair. With the molecule B, there is significant disorder in most of the atoms of the cyclohexane ring, with atoms C4B and C7B [site occupancy factor = 0.73(1)]

having alternative 'flipped' chair conformational sites [C4C and C7C: S.O.F. = 0.27(1)], C having the [C8(*R*),C9(*S*)] configuration. The two ring systems are also different with respect to the rotation about the N-C(phenyl) bond as indicated previously and both this and the disorder are therefore responsible for the presence of the two independent pseudo-racemic molecules in the $P2_1$ asymmetric unit rather than forming the common centrosymmetrically-related recemic pairs found in most examples [13,14].

INSERT 4:

Figure 3 (Atom numbering scheme for molecule A of compound 2)

(CHDC33.TIF)

Figure 4 (Atom numbering scheme for molecule B of compound 2)

CHDC34.TIF)

There are only minor intermolecular aromatic C---H...O interactions in the crystal structure [C21B-H...O1B^{i}, 3.153(4) Å, and C61A-H...O3A^{ii}, 3.058(3) Å; symmetry codes: (i) *x*, *y* - 1, *z*; (ii) *x*, *y* + 1, *z*].

The cyclic imide **3** also has disorder in the cyclohexane moiety of the molecule which is more extensive than in **2**, involving most of the perhydroisoindole-1,3-dione atoms (Fig. 5). Both components have the *cis*-configuration with the major component [site occupancy factor = 0.85(1) having a slightly distorted chair conformation while the minor component adopts a more distorted chair conformation. The minimum torsion angle between the benzene ring and the isoindoline ring (C21-C11-N1-C1) is -65.7(3)^{o}, while the carboxylic acid group is essentially coplanar with the benzene ring [torsion angle C21-C31-C311-O32, 175.2(2)^{o}]. This conformation is maintained by an intramolecular hydrogen

bond between the hydroxyl and carboxyl groups [O41-H…O32, 2.603(3)^{o}; O-H…O angle 138^{o}], which is similar to that found in the parent salicylic acid [22].

INSERT 5:

Figure 5 (Atom numbering scheme for compound 3) (CHDC35.TIF)

In the crystal packing (Fig. 6), the molecules form one-dimensional zigzag chains through intermolecular carboxylic acid O-H…O(isoindoline) hydrogen bonds [O31-H…O1^{i}, 2.707(3) Å: O-H…O angle 179^{o}: symmetry code (i) -*x*, *y* – ½, -*z* + ½]. These chains extend across the *b* cell direction in the unit cell.

INSERT 6:

Figure 6. (Hydrogen bonding of 3 in the unit cell) (CHDC36.TIF)

Conclusion

This work provides further examples of the structures of cyclic imides and open-chain amide carboxylic acids from the facile reaction of *cis*-cyclohexane-1,2-dicarboxylic anhydride with substituted anilines. Within this relatively small set of known structures (currently 11 examples, including **1-3**), there is a much higher incidence of cyclic imides (8) compared to amide acids (3). However, functional group influence upon which form is preferred is not apparent from the examples structurally characterized in this and previous work with this series of compounds.

Supplementary material

CCDC entries 862423, 862424 and 862425 contain the supplementary crystallographic data for compounds **3, 2** and **1,** respectively**,** from this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* http://www.ccdc.cam.ac.uk/data_request/cif or by e-mailing

data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK.

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Table 1. Crystal data for compounds **1-3**.

Compound	**1**	**2**	**3**
CCDC reference	862425	862424	862423
Molecular formula	$C_{14}H_{16}ClNO_3$	$C_{14}H_{14}BrNO_2$	$C_{15}H_{15}NO_5$
M_r	281.73	308.16	289.28
Crystal system	orthorhombic	monoclinic	orthorhombic
Space group	*Pbcn*	$P2_1$	$P2_12_12_1$
a (Å)	20.1753(10)	11.5321(3)	6.4642(3)
b (Å)	8.6267(4)	6.7095(2)	12.8196(5)
c (Å)	15.9940(9)	17.2040(5)	16.4197(7)
β (°)	90	102.527(3)	90
V (Å^3)	2783.7(2)	1299.46(7)	1360.68(10)
Z	8	4	4
D_c (g cm^{-3})	1.344	1.568	1.412
μ (mm^{-1})	0.278	3.156	0.107
F(000)	1184	1288	608
Reflections total, θ_{max} (°)	9279, 26.0	9056, 26.0	5237, 28.9
Crystal size (mm)	0.38 x 0.22 x 0.10	0.40 x 0.30 x 0.05	0.35 x 0.10 x 0.08
Collection range: *h* *k* *l*	 -15 to 24 -10 to 6 -12 to 19	 -11 to 14 -8 to 8 -21 to 21	 -5 to 8 -16 to 16 -20 to 17
Reflections (independent)	2735	5041	1818
Reflections [$F^2>2\sigma(F^2)$]	1616	3774	1624
R_{int}	0.0527	0.0236	0.0264
$R1$ [a] [$F^2>2\sigma(F^2)$]	0.0609	0.0299	0.0433
$wR2$ [a] (all data)	0.1412	0.0513	0.1011
S [a]	0.94	0.83	1.10
n_p	172	333	222
Residuals (max/min) (eÅ^{-3})	0.299/-0.222	0.400/-0.370	0.175/-0.173
Transmission factors (max/min)	0.880/0.980	0.714/0.980	0.969/0.990

[a] $R1 = (\Sigma\,||F_o| - |F_c||)/\Sigma\,|F_o|)$; $wR2 = \{\Sigma\,[w(F_o^2 - F_c^2)^2] / \Sigma\,[w(F_o^2)^2]\}^{½}$; $S = \{\Sigma\,[w(F_o^2 - F_c^2)^2] / (n\text{-}p)\}^{½}$.

Figures

Figure 1. Atom numbering scheme for **1,** with non-hydrogen atoms shown as 40% probability ellipsoids [15].

Figure 2. The hydrogen-bonded chain structures in **1** showing the head-to-head cyclic dimeric carboxylic acid hydrogen-bonding associations and the extension through N-H…O hydrogen bonds. Non-associative H-atoms are omitted.

Figure 3. The ordered (A) molecule in the asymmetric unit of **2,** showing 40% probability ellipsoids.

Figure 4. The partially disordered (B) molecule in the asymmetric unit of **2,** showing 30% probability ellipsoids.

Figure 5. The molecular conformation and atom numbering scheme for the disordered molecule of **3**, showing 30% probability ellipsoids

Figure 6. A perspective view of **3** in the unit cell viewed down the *a* axial direction. The minor disordered component (A) of the hexahydroisoindole ring system and non-associative H-atoms are omitted.

Supplementary Material (not for publication)

CCDC deposited CIF entries for compounds (1)-(3)

```
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# Reference: Smith & Wermuth (2012). J. Chem. Cryst.
# Compound 1
# CCDC 862425
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 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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_computing_data_reduction        'CrysAlis PRO'
_computing_structure_solution    'SIR92 (Altomare, 1994)'
_computing_structure_refinement
'SHELXL97 (Sheldrick, 2008) within WinGX (Farrugia, 1999)'
_computing_molecular_graphics     'PLATON (Spek, 2009)'
_computing_publication_material   'PLATON'

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0765P)^2^] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     constr
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_number_reflns          2735
_refine_ls_number_parameters      172
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.1100
_refine_ls_R_factor_gt            0.0609
_refine_ls_wR_factor_ref          0.1412
_refine_ls_wR_factor_gt           0.1253
_refine_ls_goodness_of_fit_ref    0.942
_refine_ls_restrained_S_all       0.942
_refine_ls_shift/su_max           0.000
_refine_ls_shift/su_mean          0.000
_refine_diff_density_max    0.299
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_atom_site_fract_z
_atom_site_occupancy
_atom_site_U_iso_or_equiv
_atom_site_calc_flag
_atom_site_refinement_flags
Cl41 Cl Uani 0.96433(5) 0.59159(12) 0.34777(6) 1.000 0.0708(4) . .
```

```
O12 O Uani 0.68552(9) 0.5156(2) 0.57974(14) 1.000 0.0423(7) . .
O21 O Uani 0.55529(11) 0.3496(2) 0.51992(14) 1.000 0.0496(8) . .
O22 O Uani 0.52008(10) 0.5434(2) 0.60053(13) 1.000 0.0514(8) . .
N11 N Uani 0.75798(11) 0.3186(3) 0.56445(16) 1.000 0.0361(8) . .
C1 C Uani 0.65304(14) 0.2674(3) 0.63534(18) 1.000 0.0361(9) . .
C2 C Uani 0.58928(14) 0.3503(3) 0.66177(18) 1.000 0.0379(10) . .
C3 C Uani 0.60015(15) 0.4613(3) 0.73501(19) 1.000 0.0456(11) . .
C4 C Uani 0.63172(18) 0.3795(4) 0.8088(2) 1.000 0.0575(11) . .
C5 C Uani 0.69575(16) 0.2992(4) 0.7826(2) 1.000 0.0543(12) . .
C6 C Uani 0.68429(15) 0.1876(3) 0.7114(2) 1.000 0.0483(11) . .
C11 C Uani 0.80642(13) 0.3935(3) 0.51465(17) 1.000 0.0332(9) . .
C12 C Uani 0.69999(13) 0.3795(3) 0.59100(17) 1.000 0.0326(9) . .
C21 C Uani 0.79068(14) 0.5049(3) 0.45607(18) 1.000 0.0388(9) . .
C22 C Uani 0.55340(13) 0.4224(3) 0.58966(19) 1.000 0.0373(10) . .
C31 C Uani 0.83914(15) 0.5677(3) 0.40571(19) 1.000 0.0438(10) . .
C41 C Uani 0.90401(15) 0.5175(3) 0.41448(19) 1.000 0.0437(10) . .
C51 C Uani 0.92066(15) 0.4079(3) 0.47253(19) 1.000 0.0435(10) . .
C61 C Uani 0.87208(14) 0.3465(3) 0.5233(2) 1.000 0.0407(10) . .
H1 H Uiso 0.64080 0.18650 0.59520 1.000 0.0430 calc R
H2 H Uiso 0.55980 0.26890 0.68290 1.000 0.0450 calc R
H3A H Uiso 0.62850 0.54590 0.71720 1.000 0.0550 calc R
H3B H Uiso 0.55790 0.50480 0.75200 1.000 0.0550 calc R
H4A H Uiso 0.60110 0.30340 0.83120 1.000 0.0690 calc R
H4B H Uiso 0.64120 0.45430 0.85250 1.000 0.0690 calc R
H5A H Uiso 0.72790 0.37660 0.76570 1.000 0.0650 calc R
H5B H Uiso 0.71390 0.24330 0.83000 1.000 0.0650 calc R
H6A H Uiso 0.65540 0.10470 0.73010 1.000 0.0580 calc R
H6B H Uiso 0.72630 0.14180 0.69510 1.000 0.0580 calc R
H11 H Uiso 0.77500 0.22600 0.56900 1.000 0.0430 . R
H21 H Uiso 0.74700 0.53790 0.45050 1.000 0.0470 calc R
H22 H Uiso 0.52600 0.38000 0.47400 1.000 0.0620 . R
H31 H Uiso 0.82850 0.64290 0.36630 1.000 0.0530 calc R
H51 H Uiso 0.96440 0.37510 0.47780 1.000 0.0520 calc R
H61 H Uiso 0.88320 0.27320 0.56350 1.000 0.0490 calc R

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_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
Cl41 0.0475(6) 0.1086(8) 0.0562(6) 0.0048(5) 0.0163(5) -0.0214(5)
O12 0.0282(11) 0.0299(11) 0.0688(15) 0.0090(9) 0.0073(11) 0.0039(8)
O21 0.0411(13) 0.0568(13) 0.0508(14) -0.0009(11) -0.0106(11) 0.0038(10)
O22 0.0369(12) 0.0623(13) 0.0549(14) 0.0022(11) -0.0044(11) 0.0142(10)
N11 0.0268(14) 0.0272(13) 0.0544(16) 0.0061(11) 0.0013(12) 0.0019(11)
C1 0.0273(16) 0.0345(15) 0.0465(18) 0.0050(13) -0.0012(14) -0.0038(11)
C2 0.0267(16) 0.0381(16) 0.0488(18) 0.0101(13) 0.0014(14) -0.0075(12)
C3 0.0355(19) 0.0573(18) 0.0440(18) 0.0004(15) 0.0005(15) 0.0028(14)
C4 0.055(2) 0.070(2) 0.0476(19) 0.0054(17) -0.0033(18) -0.0027(17)
C5 0.048(2) 0.062(2) 0.053(2) 0.0167(17) -0.0133(17) -0.0035(16)
C6 0.0344(18) 0.0476(17) 0.063(2) 0.0176(15) 0.0014(16) -0.0009(14)
C11 0.0279(16) 0.0290(14) 0.0426(16) -0.0031(12) -0.0014(13) -0.0017(11)
C12 0.0245(15) 0.0331(16) 0.0401(16) -0.0003(12) -0.0044(12) 0.0013(11)
C21 0.0292(16) 0.0444(16) 0.0429(17) -0.0011(14) -0.0021(14) 0.0007(13)
C22 0.0210(15) 0.0423(17) 0.0486(19) 0.0024(15) 0.0013(14) -0.0050(13)
C31 0.0405(19) 0.0538(18) 0.0371(17) 0.0071(14) -0.0024(15) -0.0030(14)
C41 0.0349(18) 0.0543(18) 0.0419(18) -0.0087(15) 0.0081(15) -0.0102(14)
C51 0.0279(17) 0.0466(17) 0.0560(19) -0.0066(16) 0.0007(15) -0.0007(13)
```

```
C61 0.0284(17) 0.0388(16) 0.0549(19) 0.0036(13) -0.0014(15) 0.0037(13)

_geom_special_details
;
 Bond distances, angles etc. have been calculated using the
 rounded fractional coordinates. All su's are estimated
 from the variances of the (full) variance-covariance matrix.
 The cell esds are taken into account in the estimation of
 distances, angles and torsion angles
;

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 O12     C12        1.223(3)                    .       .                 no
 O21     C22        1.281(4)                    .       .                 no
 O22     C22        1.254(3)                    .       .                 no
 O21     H22          0.98                    .       .                 no
 N11     C11        1.417(4)                    .       .                 no
 N11     C12        1.351(4)                    .       .                 no
 N11     H11         0.87                     .       .                 no
 C1      C12        1.528(4)                    .       .                 no
 C1      C6         1.533(4)                    .       .                 no
 C1      C2         1.531(4)                    .       .                 no
 C2      C3         1.529(4)                    .       .                 no
 C2      C22        1.497(4)                    .       .                 no
 C3      C4         1.515(4)                    .       .                 no
 C4      C5         1.525(5)                    .       .                 no
 C5      C6         1.509(4)                    .       .                 no
 C11     C61        1.392(4)                    .       .                 no
 C11     C21        1.379(4)                    .       .                 no
 C21     C31        1.378(4)                    .       .                 no
 C31     C41        1.386(4)                    .       .                 no
 C41     C51        1.367(4)                    .       .                 no
 C51     C61        1.379(4)                    .       .                 no
 C1      H1           0.9800                    .       .                 no
 C2      H2           0.9800                    .       .                 no
 C3      H3A          0.9700                    .       .                 no
 C3      H3B          0.9700                    .       .                 no
 C4      H4A          0.9700                    .       .                 no
 C4      H4B          0.9700                    .       .                 no
 C5      H5A          0.9700                    .       .                 no
 C5      H5B          0.9700                    .       .                 no
 C6      H6A          0.9700                    .       .                 no
 C6      H6B          0.9700                    .       .                 no
 C21     H21          0.9300                    .       .                 no
 C31     H31          0.9300                    .       .                 no
 C51     H51          0.9300                    .       .                 no
 C61     H61          0.9300                    .       .                 no

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_geom_angle_site_symmetry_1
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_geom_angle_site_symmetry_3
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 C22     O21     H22          120              .       .       .          no
 C11     N11     C12        126.6(2)           .       .       .          no
 C12     N11     H11          132              .       .       .          no
 C11     N11     H11          101              .       .       .          no
 C2      C1      C12        110.7(2)           .       .       .          no
 C6      C1      C12        113.4(2)           .       .       .          no
 C2      C1      C6         109.6(2)           .       .       .          no
 C1      C2      C3         112.6(2)           .       .       .          no
 C1      C2      C22        112.8(2)           .       .       .          no
 C3      C2      C22        113.5(2)           .       .       .          no
 C2      C3      C4         111.4(2)           .       .       .          no
 C3      C4      C5         110.7(3)           .       .       .          no
 C4      C5      C6         111.6(3)           .       .       .          no
 C1      C6      C5         112.0(2)           .       .       .          no
 N11     C11     C21        122.7(2)           .       .       .          no
 N11     C11     C61        117.9(2)           .       .       .          no
 C21     C11     C61        119.3(3)           .       .       .          no
 N11     C12     C1         115.9(2)           .       .       .          no
 O12     C12     N11        122.3(2)           .       .       .          no
 O12     C12     C1         121.9(2)           .       .       .          no
 C11     C21     C31        120.5(3)           .       .       .          no
 O21     C22     O22        123.0(3)           .       .       .          no
 O21     C22     C2         116.9(2)           .       .       .          no
 O22     C22     C2         119.9(3)           .       .       .          no
 C21     C31     C41        119.2(3)           .       .       .          no
 Cl41    C41     C31        118.9(2)           .       .       .          no
 C31     C41     C51        121.1(3)           .       .       .          no
 Cl41    C41     C51        119.9(2)           .       .       .          no
 C41     C51     C61        119.4(3)           .       .       .          no
 C11     C61     C51        120.4(3)           .       .       .          no
 C2      C1      H1           108.00           .       .       .          no
 C6      C1      H1           108.00           .       .       .          no
 C12     C1      H1           108.00           .       .       .          no
 C1      C2      H2           106.00           .       .       .          no
 C3      C2      H2           106.00           .       .       .          no
 C22     C2      H2           106.00           .       .       .          no
 C2      C3      H3A          109.00           .       .       .          no
 C2      C3      H3B          109.00           .       .       .          no
 C4      C3      H3A          109.00           .       .       .          no
 C4      C3      H3B          109.00           .       .       .          no
 H3A     C3      H3B          108.00           .       .       .          no
 C3      C4      H4A          110.00           .       .       .          no
 C3      C4      H4B          110.00           .       .       .          no
 C5      C4      H4A          109.00           .       .       .          no
 C5      C4      H4B          109.00           .       .       .          no
 H4A     C4      H4B          108.00           .       .       .          no
 C4      C5      H5A          109.00           .       .       .          no
 C4      C5      H5B          109.00           .       .       .          no
 C6      C5      H5A          109.00           .       .       .          no
 C6      C5      H5B          109.00           .       .       .          no
 H5A     C5      H5B          108.00           .       .       .          no
 C1      C6      H6A          109.00           .       .       .          no
 C1      C6      H6B          109.00           .       .       .          no
 C5      C6      H6A          109.00           .       .       .          no
 C5      C6      H6B          109.00           .       .       .          no
 H6A     C6      H6B          108.00           .       .       .          no
 C11     C21     H21          120.00           .       .       .          no
 C31     C21     H21          120.00           .       .       .          no
```

```
 C21     C31     H31          120.00             .       .       .          no
 C41     C31     H31          120.00             .       .       .          no
 C41     C51     H51          120.00             .       .       .          no
 C61     C51     H51          120.00             .       .       .          no
 C11     C61     H61          120.00             .       .       .          no
 C51     C61     H61          120.00             .       .       .          no

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 C12     N11     C11     C21        -30.8(4)     .       .       .       .   no
 C12     N11     C11     C61        152.1(3)     .       .       .       .   no
 C11     N11     C12     O12         -6.4(4)     .       .       .       .   no
 C11     N11     C12     C1         173.0(3)     .       .       .       .   no
 C6      C1      C2      C3          53.7(3)     .       .       .       .   no
 C6      C1      C2      C22       -176.2(2)     .       .       .       .   no
 C12     C1      C2      C3         -72.1(3)     .       .       .       .   no
 C12     C1      C2      C22         58.0(3)     .       .       .       .   no
 C2      C1      C6      C5         -54.8(3)     .       .       .       .   no
 C12     C1      C6      C5          69.5(3)     .       .       .       .   no
 C2      C1      C12     O12         -0.3(4)     .       .       .       .   no
 C2      C1      C12     N11       -179.7(2)     .       .       .       .   no
 C6      C1      C12     O12       -124.0(3)     .       .       .       .   no
 C6      C1      C12     N11         56.6(3)     .       .       .       .   no
 C1      C2      C3      C4         -54.8(3)     .       .       .       .   no
 C22     C2      C3      C4         175.5(2)     .       .       .       .   no
 C1      C2      C22     O21         35.4(3)     .       .       .       .   no
 C1      C2      C22     O22       -148.9(2)     .       .       .       .   no
 C3      C2      C22     O21        165.0(2)     .       .       .       .   no
 C3      C2      C22     O22        -19.3(4)     .       .       .       .   no
 C2      C3      C4      C5          54.7(3)     .       .       .       .   no
 C3      C4      C5      C6         -56.1(4)     .       .       .       .   no
 C4      C5      C6      C1          56.8(3)     .       .       .       .   no
 N11     C11     C21     C31       -176.1(3)     .       .       .       .   no
 C61     C11     C21     C31          0.9(4)     .       .       .       .   no
 N11     C11     C61     C51        175.7(3)     .       .       .       .   no
 C21     C11     C61     C51         -1.5(4)     .       .       .       .   no
 C11     C21     C31     C41          0.1(4)     .       .       .       .   no
 C21     C31     C41     Cl41       177.4(2)     .       .       .       .   no
 C21     C31     C41     C51         -0.5(4)     .       .       .       .   no
 Cl41    C41     C51     C61       -178.0(2)     .       .       .       .   no
 C31     C41     C51     C61         -0.1(4)     .       .       .       .   no
 C41     C51     C61     C11          1.1(4)     .       .       .       .   no


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_geom_hbond_distance_HA
_geom_hbond_distance_DA
_geom_hbond_angle_DHA
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  _geom_hbond_publ_flag
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  #D   H   A   D - H  H...A   D...A    D - H...A  symm(A)
  #
  N11   H11  O12    0.87     1.99   2.862(3)     180   7_645 yes
  O21   H22  O22    0.98     1.65   2.622(3)     172   5_666 yes
  C3    H3A  O12    0.97     2.50   3.058(4)     117     .   no
  C21   H21  O12    0.93     2.42   2.902(3)     112     .   no

  # END of Data for (1)

#------------------------------------------------------------------------------
# Reference: Smith & Wermuth (2012). J. Chem. Cryst.
# Compound 2
# CCDC 862424
#------------------------------------------------------------------------------

data_gs11gs2_CHDCBRAN

_audit_creation_method            SHELXL97
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;
2-(4-bromophenyl)-3a,4,5,6,7,7a-hexahydroisoindole-1,3-dione
;
_chemical_name_common
;
2-(4-bromophenyl)perhydroisoindole-1,3-dione
;
_chemical_melting_point           ?
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 _atom_type_scat_dispersion_imag
 _atom_type_scat_source
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 'H'  'H'   0.0000   0.0000
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 'N'  'N'   0.0061   0.0033
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0106   0.0060
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 'Br'  'Br'  -0.2901   2.4595
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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_symmetry_space_group_name_Hall  'P 2yb'

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 '-x, y+1/2, -z'
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_cell_length_c                    17.2040(5)
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_cell_angle_beta                  102.527(3)
_cell_angle_gamma                 90.00
_cell_volume                      1299.46(7)
_cell_formula_units_Z             4
_cell_measurement_temperature     200(2)
_cell_measurement_reflns_used     4567
_cell_measurement_theta_min       3.26
_cell_measurement_theta_max       28.74

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;
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;

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'Oxford Diffraction Gemini-S CCD-detector diffractometer'
_diffrn_measurement_method       '\w scans'
_diffrn_detector_area_resol_mean  16.08
_diffrn_standards_number          0
_diffrn_standards_interval_count  .
_diffrn_standards_interval_time   .
_diffrn_standards_decay_%         ?
_diffrn_reflns_number             9056
_diffrn_reflns_av_R_equivalents   0.0236
_diffrn_reflns_av_sigmaI/netI     0.0578
_diffrn_reflns_limit_h_min        -11
_diffrn_reflns_limit_h_max        14
_diffrn_reflns_limit_k_min        -8
_diffrn_reflns_limit_k_max        8
_diffrn_reflns_limit_l_min        -21
_diffrn_reflns_limit_l_max        21
_diffrn_reflns_theta_min          3.27
_diffrn_reflns_theta_max          26.00
_diffrn_measured_fraction_theta_max    0.998
_diffrn_reflns_theta_full              26.00
_diffrn_measured_fraction_theta_full   0.998

_reflns_number_total              5041
```

```
_reflns_number_gt                3774
_reflns_threshold_expression     'I>2\s(I)'

_computing_data_collection       'CrysAlis PRO'
_computing_cell_refinement       'CrysAlis PRO'
_computing_data_reduction        'CrysAlis PRO'
_computing_structure_solution  'SIR92 (Altomare <i>et al.</i>, 1994)'
_computing_structure_refinement
 'SHELXL97 (Sheldrick, 2008) within WinGX (Farrugia, 1999)'
_computing_molecular_graphics    'PLATON  (Spek, 2009)'
_computing_publication_material  'PLATON'

_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0243P)^2^] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     constr
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_abs_structure_details  'Flack, 1983: 2249 Friedel pairs'
_refine_ls_abs_structure_Flack    0.047(6)
_refine_ls_number_reflns          5041
_refine_ls_number_parameters      333
_refine_ls_number_restraints      1
_refine_ls_R_factor_all           0.0447
_refine_ls_R_factor_gt            0.0299
_refine_ls_wR_factor_ref          0.0513
_refine_ls_wR_factor_gt           0.0496
_refine_ls_goodness_of_fit_ref    0.833
_refine_ls_restrained_S_all       0.833
_refine_ls_shift/su_max           0.001
_refine_ls_shift/su_mean          0.000
_refine_diff_density_max    0.400
_refine_diff_density_min   -0.370
_refine_diff_density_rms    0.045

loop_
_atom_site_label
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_atom_site_thermal_displace_type
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_occupancy
_atom_site_U_iso_or_equiv
_atom_site_calc_flag
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```
_atom_site_refinement_flags
Br4B Br Uani 0.52220(3) 0.20543(6) 0.61041(2) 1.000 0.0706(2) . .
O1B O Uani 0.1285(2) 0.9154(3) 0.68904(13) 1.000 0.0499(8) . .
O3B O Uani -0.0549(2) 0.4101(4) 0.53254(15) 1.000 0.0755(10) . .
N2B N Uani 0.0620(2) 0.6400(3) 0.61224(14) 1.000 0.0337(8) . .
C1B C Uani 0.0492(3) 0.8254(5) 0.64669(17) 1.000 0.0374(10) . .
C3B C Uani -0.0449(3) 0.5669(5) 0.56862(19) 1.000 0.0476(12) . .
C4B C Uani -0.2077(5) 0.5940(7) 0.6341(3) 0.730 0.054(2) . .
C5B C Uani -0.2811(4) 0.7190(8) 0.6701(2) 1.000 0.0914(19) . .
C6B C Uani -0.2091(4) 0.8760(9) 0.7175(2) 1.000 0.105(2) . .
C7B C Uani -0.1387(4) 1.0095(6) 0.6680(3) 0.730 0.063(2) . .
C8B C Uani -0.0784(3) 0.8896(5) 0.6189(2) 1.000 0.0576(14) . .
C9B C Uani -0.1417(3) 0.7089(6) 0.57751(18) 1.000 0.0458(11) . .
C11B C Uani 0.1729(3) 0.5415(4) 0.61525(17) 1.000 0.0323(10) . .
C21B C Uani 0.1915(3) 0.3546(5) 0.64779(15) 1.000 0.0389(10) . .
C31B C Uani 0.2962(3) 0.2549(4) 0.64679(17) 1.000 0.0420(11) . .
C41B C Uani 0.3808(3) 0.3444(6) 0.61315(16) 1.000 0.0418(10) . .
C51B C Uani 0.3631(3) 0.5340(5) 0.58108(18) 1.000 0.0450(12) . .
C61B C Uani 0.2583(3) 0.6326(4) 0.58154(16) 1.000 0.0370(11) . .
C4C C Uiso -0.2593(4) 0.6550(6) 0.5875(3) 0.270 0.047(3) . .
C7C C Uiso -0.0821(4) 0.8654(6) 0.7037(3) 0.270 0.064(3) . .
Br4A Br Uani -0.30253(3) 1.34323(4) 0.86437(2) 1.000 0.0476(1) . .
O1A O Uani 0.30063(16) 1.2101(3) 0.97971(11) 1.000 0.0394(7) . .
O3A O Uani 0.12055(19) 0.6670(3) 0.84468(13) 1.000 0.0433(8) . .
N2A N Uani 0.1839(2) 0.9538(3) 0.91552(13) 1.000 0.0274(8) . .
C1A C Uani 0.2908(3) 1.0471(4) 0.94919(16) 1.000 0.0303(10) . .
C3A C Uani 0.2001(3) 0.7646(4) 0.88403(18) 1.000 0.0314(11) . .
C4A C Uani 0.3815(3) 0.5755(4) 0.85802(18) 1.000 0.0397(11) . .
C5A C Uani 0.4194(3) 0.6828(4) 0.79091(17) 1.000 0.0416(11) . .
C6A C Uani 0.4998(3) 0.8579(5) 0.82214(18) 1.000 0.0441(10) . .
C7A C Uani 0.4299(3) 1.0063(5) 0.8613(2) 1.000 0.0417(11) . .
C8A C Uani 0.3892(3) 0.9137(4) 0.93191(17) 1.000 0.0315(10) . .
C9A C Uani 0.3290(2) 0.7108(4) 0.91270(16) 1.000 0.0326(10) . .
C11A C Uani 0.0695(2) 1.0473(4) 0.90664(16) 1.000 0.0275(9) . .
C21A C Uani -0.0218(3) 0.9500(4) 0.93159(16) 1.000 0.0305(10) . .
C31A C Uani -0.1328(3) 1.0375(4) 0.92004(16) 1.000 0.0335(10) . .
C41A C Uani -0.1492(2) 1.2217(5) 0.88464(15) 1.000 0.0311(9) . .
C51A C Uani -0.0587(2) 1.3225(5) 0.86158(15) 1.000 0.0308(9) . .
C61A C Uani 0.0518(3) 1.2335(4) 0.87262(16) 1.000 0.0319(9) . .
H21B H Uiso 0.13400 0.29500 0.67050 1.000 0.0470 calc R
H31B H Uiso 0.30930 0.12810 0.66870 1.000 0.0510 calc R
H41B H Uiso -0.26280 0.50570 0.59980 0.730 0.0650 . R
H41C H Uiso -0.26920 0.51180 0.58170 0.270 0.0560 . R
H42B H Uiso -0.15390 0.51100 0.67180 0.730 0.0650 . R
H42C H Uiso -0.31820 0.71780 0.54590 0.270 0.0560 . R
H51B H Uiso  0.42150 0.59470 0.55930 1.000 0.0540 . R
H52B H Uiso -0.31480 0.63470 0.70530 1.000 0.1100 . R
H53B H Uiso -0.34580 0.78420 0.63350 1.000 0.1100 . R
H61B H Uiso  0.24490 0.75920 0.55940 1.000 0.0440 . R
H62B H Uiso -0.14810 0.80870 0.75640 1.000 0.1260 . R
H63B H Uiso -0.25570 0.95600 0.74650 1.000 0.1260 . R
H71B H Uiso -0.19700 1.09540 0.63500 0.730 0.0760 . R
H72B H Uiso -0.08150 1.09320 0.70310 0.730 0.0760 . R
H8B H Uiso -0.07550 0.97970 0.57450 1.000 0.0700 . R
H9B H Uiso -0.19120 0.74450 0.52560 1.000 0.0550 . R
H71C H Uiso -0.03450 0.96910 0.73460 0.270 0.0770 . R
H72C H Uiso -0.04740 0.73780 0.72250 0.270 0.0770 . R
H8A H Uiso 0.45530 0.90460 0.97840 1.000 0.0380 calc R
H9A H Uiso 0.33600 0.64000 0.96330 1.000 0.0390 calc R
H21A H Uiso -0.00860 0.82600 0.95610 1.000 0.0370 calc R
H31A H Uiso -0.19510 0.97290 0.93590 1.000 0.0400 calc R
```

```
H42A H Uiso 0.44970 0.50680 0.88950 1.000 0.0480 calc R
H43A H Uiso 0.32300 0.47540 0.83570 1.000 0.0480 calc R
H51A H Uiso -0.07150 1.44860 0.83890 1.000 0.0370 calc R
H52A H Uiso 0.34970 0.73060 0.75330 1.000 0.0500 calc R
H53A H Uiso 0.46130 0.59120 0.76310 1.000 0.0500 calc R
H61A H Uiso 0.11400 1.29930 0.85710 1.000 0.0380 calc R
H62A H Uiso 0.52680 0.92160 0.77860 1.000 0.0530 calc R
H63A H Uiso 0.56880 0.81160 0.86060 1.000 0.0530 calc R
H71A H Uiso 0.36110 1.05150 0.82230 1.000 0.0500 calc R
H72A H Uiso 0.47940 1.12130 0.87930 1.000 0.0500 calc R

loop_
_atom_site_aniso_label
_atom_site_aniso_U_11
_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
Br4B 0.0541(3) 0.0795(3) 0.0810(3) 0.0049(2) 0.0210(2) 0.0208(2)
O1B 0.0506(15) 0.0314(12) 0.0626(15) -0.0062(10) 0.0009(12) -0.0094(10)
O3B 0.0569(17) 0.0734(18) 0.0869(19) -0.0502(15) -0.0050(13) 0.0006(13)
N2B 0.0335(15) 0.0330(14) 0.0331(14) -0.0028(11) 0.0037(12) -0.0022(11)
C1B 0.050(2) 0.0288(17) 0.0332(17) 0.0072(17) 0.0089(15) -0.0037(18)
C3B 0.047(2) 0.049(2) 0.044(2) -0.0103(18) 0.0040(17) -0.0048(17)
C4B 0.058(4) 0.036(3) 0.070(4) 0.005(3) 0.017(3) 0.008(3)
C5B 0.065(3) 0.144(4) 0.076(3) -0.004(3) 0.039(2) 0.007(4)
C6B 0.099(4) 0.155(5) 0.057(3) -0.037(3) 0.011(3) 0.045(4)
C7B 0.055(4) 0.045(3) 0.088(4) -0.030(3) 0.014(3) 0.003(3)
C8B 0.042(2) 0.045(2) 0.085(3) 0.0049(19) 0.012(2) -0.0028(17)
C9B 0.0319(18) 0.067(2) 0.0363(18) -0.0099(18) 0.0028(14) 0.0019(19)
C11B 0.0336(19) 0.0298(16) 0.0312(17) -0.0012(14) 0.0017(14) -0.0013(14)
C21B 0.0491(19) 0.0325(16) 0.0359(16) 0.0006(17) 0.0110(14) -0.0090(19)
C31B 0.051(2) 0.0338(17) 0.040(2) 0.0044(14) 0.0073(17) 0.0039(17)
C41B 0.0432(18) 0.0483(18) 0.0323(16) -0.0026(19) 0.0047(14) 0.005(2)
C51B 0.042(2) 0.057(2) 0.037(2) 0.0050(17) 0.0105(16) -0.0076(18)
C61B 0.041(2) 0.0376(18) 0.0326(18) 0.0085(13) 0.0084(15) -0.0027(15)
Br4A 0.0312(2) 0.0566(2) 0.0556(2) 0.0006(2) 0.0107(1) 0.0058(2)
O1A 0.0396(12) 0.0335(10) 0.0448(12) -0.0159(11) 0.0087(9) 0.0004(11)
O3A 0.0427(14) 0.0232(12) 0.0652(15) -0.0058(11) 0.0144(12) -0.0103(10)
N2A 0.0292(15) 0.0212(12) 0.0335(15) -0.0022(11) 0.0103(11) -0.0026(11)
C1A 0.0326(18) 0.0288(16) 0.0300(17) 0.0008(14) 0.0077(14) 0.0035(14)
C3A 0.037(2) 0.0233(16) 0.0370(19) 0.0069(13) 0.0146(16) -0.0013(13)
C4A 0.041(2) 0.0262(17) 0.052(2) -0.0061(16) 0.0101(17) 0.0068(14)
C5A 0.044(2) 0.0447(19) 0.0351(17) -0.0065(16) 0.0062(15) 0.0105(17)
C6A 0.0415(18) 0.0445(17) 0.0510(18) 0.0001(19) 0.0205(15) -0.0004(19)
C7A 0.038(2) 0.0305(17) 0.061(2) -0.0063(15) 0.0204(17) -0.0046(15)
C8A 0.0260(17) 0.0332(16) 0.0328(17) -0.0041(12) 0.0009(13) 0.0039(12)
C9A 0.0373(18) 0.0252(15) 0.0350(17) 0.0012(15) 0.0073(14) 0.0056(16)
C11A 0.0282(17) 0.0251(15) 0.0310(16) -0.0009(13) 0.0105(13) -0.0013(13)
C21A 0.040(2) 0.0237(14) 0.0277(17) 0.0019(13) 0.0070(14) -0.0053(14)
C31A 0.0346(19) 0.0344(17) 0.0336(18) 0.0031(14) 0.0121(14) -0.0080(15)
C41A 0.0216(16) 0.0393(17) 0.0313(16) -0.0072(15) 0.0034(12) 0.0039(15)
C51A 0.0340(17) 0.0240(15) 0.0346(15) 0.0023(14) 0.0076(13) -0.0033(16)
C61A 0.0330(17) 0.0246(15) 0.0421(17) 0.0010(14) 0.0170(14) -0.0053(14)

_geom_special_details
;
 Bond distances, angles etc. have been calculated using the
 rounded fractional coordinates. All su's are estimated
 from the variances of the (full) variance-covariance matrix.
```

```
 The cell esds are taken into account in the estimation of
 distances, angles and torsion angles
;

loop_
_geom_bond_atom_site_label_1
_geom_bond_atom_site_label_2
_geom_bond_distance
_geom_bond_site_symmetry_1
_geom_bond_site_symmetry_2
_geom_bond_publ_flag
 Br4B    C41B       1.888(4)                    .      .                 no
 Br4A    C41A       1.909(3)                    .      .                 no
 O1B     C1B        1.201(4)                    .      .                 no
 O3B     C3B        1.214(4)                    .      .                 no
 O1A     C1A        1.208(3)                    .      .                 no
 O3A     C3A        1.208(4)                    .      .                 no
 N2B     C1B        1.399(4)                    .      .                 no
 N2B     C3B        1.386(4)                    .      .                 no
 N2B     C11B       1.431(4)                    .      .                 no
 N2A     C1A        1.392(4)                    .      .                 no
 N2A     C3A        1.409(3)                    .      .                 no
 N2A     C11A       1.439(3)                    .      .                 no
 C1B     C8B        1.508(5)                    .      .                 no
 C3B     C9B        1.500(5)                    .      .                 no
 C4B     C5B        1.425(7)                    .      .                 no
 C4B     C9B        1.563(6)                    .      .                 no
 C4C     C5B        1.556(6)                    .      .                 no
 C4C     C9B        1.449(6)                    .      .                 no
 C5B     C6B        1.473(7)                    .      .                 no
 C6B     C7C        1.535(7)                    .      .                 no
 C6B     C7B        1.577(7)                    .      .                 no
 C7B     C8B        1.449(6)                    .      .                 no
 C7C     C8B        1.478(6)                    .      .                 no
 C8B     C9B        1.511(5)                    .      .                 no
 C11B    C61B       1.388(5)                    .      .                 no
 C11B    C21B       1.371(4)                    .      .                 no
 C21B    C31B       1.384(5)                    .      .                 no
 C31B    C41B       1.376(5)                    .      .                 no
 C41B    C51B       1.384(5)                    .      .                 no
 C51B    C61B       1.379(5)                    .      .                 no
 C4B     H41B         0.9700                    .      .                 no
 C4B     H42B         0.9700                    .      .                 no
 C4C     H42C         0.9700                    .      .                 no
 C4C     H41C         0.9700                    .      .                 no
 C5B     H52B         0.9700                    .      .                 no
 C5B     H53B         0.9700                    .      .                 no
 C6B     H63B         0.9700                    .      .                 no
 C6B     H62B         0.9700                    .      .                 no
 C7B     H72B         0.9700                    .      .                 no
 C7B     H71B         0.9700                    .      .                 no
 C7C     H71C         0.9700                    .      .                 no
 C7C     H72C         0.9700                    .      .                 no
 C8B     H8B          0.9800                    .      .                 no
 C9B     H9B          0.9800                    .      .                 no
 C21B    H21B         0.9300                    .      .                 no
 C31B    H31B         0.9300                    .      .                 no
 C51B    H51B         0.9300                    .      .                 no
 C61B    H61B         0.9300                    .      .                 no
 C1A     C8A        1.524(5)                    .      .                 no
 C3A     C9A        1.505(4)                    .      .                 no
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```
 C4A     C5A        1.504(4)                    .      .                 no
 C4A     C9A        1.524(4)                    .      .                 no
 C5A     C6A        1.521(4)                    .      .                 no
 C6A     C7A        1.527(5)                    .      .                 no
 C7A     C8A        1.526(5)                    .      .                 no
 C8A     C9A        1.531(4)                    .      .                 no
 C11A    C21A       1.384(4)                    .      .                 no
 C11A    C61A       1.376(4)                    .      .                 no
 C21A    C31A       1.383(5)                    .      .                 no
 C31A    C41A       1.373(4)                    .      .                 no
 C41A    C51A       1.373(4)                    .      .                 no
 C51A    C61A       1.383(4)                    .      .                 no
 C4A     H42A         0.9700                    .      .                 no
 C4A     H43A         0.9700                    .      .                 no
 C5A     H52A         0.9700                    .      .                 no
 C5A     H53A         0.9700                    .      .                 no
 C6A     H62A         0.9700                    .      .                 no
 C6A     H63A         0.9700                    .      .                 no
 C7A     H71A         0.9700                    .      .                 no
 C7A     H72A         0.9700                    .      .                 no
 C8A     H8A          0.9800                    .      .                 no
 C9A     H9A          0.9800                    .      .                 no
 C21A    H21A         0.9300                    .      .                 no
 C31A    H31A         0.9300                    .      .                 no
 C51A    H51A         0.9300                    .      .                 no
 C61A    H61A         0.9300                    .      .                 no

loop_
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_geom_angle_atom_site_label_2
_geom_angle_atom_site_label_3
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_geom_angle_site_symmetry_2
_geom_angle_site_symmetry_3
_geom_angle_publ_flag
 C1B     N2B     C3B        112.4(3)             .      .      .          no
 C1B     N2B     C11B       124.8(2)             .      .      .          no
 C3B     N2B     C11B       122.6(2)             .      .      .          no
 C1A     N2A     C3A        112.7(2)             .      .      .          no
 C1A     N2A     C11A       123.9(2)             .      .      .          no
 C3A     N2A     C11A       123.2(2)             .      .      .          no
 N2B     C1B     C8B        107.8(3)             .      .      .          no
 O1B     C1B     C8B        127.7(3)             .      .      .          no
 O1B     C1B     N2B        124.6(3)             .      .      .          no
 N2B     C3B     C9B        108.3(3)             .      .      .          no
 O3B     C3B     C9B        127.7(3)             .      .      .          no
 O3B     C3B     N2B        123.9(3)             .      .      .          no
 C5B     C4B     C9B        113.3(4)             .      .      .          no
 C5B     C4C     C9B        112.3(4)             .      .      .          no
 C4B     C5B     C6B        110.2(4)             .      .      .          no
 C4C     C5B     C6B        121.3(4)             .      .      .          no
 C5B     C6B     C7B        113.7(3)             .      .      .          no
 C5B     C6B     C7C        108.9(4)             .      .      .          no
 C6B     C7B     C8B        111.6(3)             .      .      .          no
 C6B     C7C     C8B        112.4(4)             .      .      .          no
 C7B     C8B     C9B        118.4(3)             .      .      .          no
 C1B     C8B     C7B        122.2(3)             .      .      .          no
 C1B     C8B     C9B        105.0(3)             .      .      .          no
 C7C     C8B     C9B        105.2(3)             .      .      .          no
 C3B     C9B     C4B        102.9(3)             .      .      .          no
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C3B	C9B	C8B	105.3(3)	.	.	.	no
C4C	C9B	C8B	120.9(3)	.	.	.	no
C3B	C9B	C4C	126.1(3)	.	.	.	no
C4B	C9B	C8B	110.7(3)	.	.	.	no
N2B	C11B	C61B	119.4(2)	.	.	.	no
N2B	C11B	C21B	119.8(3)	.	.	.	no
C21B	C11B	C61B	120.7(3)	.	.	.	no
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C21B	C31B	C41B	119.6(3)	.	.	.	no
C31B	C41B	C51B	120.8(3)	.	.	.	no
Br4B	C41B	C31B	119.4(3)	.	.	.	no
Br4B	C41B	C51B	119.8(3)	.	.	.	no
C41B	C51B	C61B	119.5(3)	.	.	.	no
C11B	C61B	C51B	119.5(3)	.	.	.	no
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C9B	C4B	H41B	105.00	.	.	.	no
C9B	C4B	H42B	112.00	.	.	.	no
C5B	C4B	H41B	105.00	.	.	.	no
H41B	C4B	H42B	107.00	.	.	.	no
H41C	C4C	H42C	108.00	.	.	.	no
C9B	C4C	H41C	109.00	.	.	.	no
C9B	C4C	H42C	109.00	.	.	.	no
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H51B	C5B	H53B	108.00	.	.	.	no
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H61B	C7C	H71C	100.00	.	.	.	no
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C6B	C7C	H72C	109.00	.	.	.	no
H71C	C7C	H72C	108.00	.	.	.	no
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C9B	C8B	H8B	103.00	.	.	.	no
C7B	C8B	H8B	102.00	.	.	.	no
C8B	C9B	H9B	111.00	.	.	.	no
C4B	C9B	H9B	115.00	.	.	.	no
C3B	C9B	H9B	111.00	.	.	.	no
C31B	C21B	H21B	120.00	.	.	.	no
C11B	C21B	H21B	120.00	.	.	.	no
C21B	C31B	H31B	120.00	.	.	.	no
C41B	C31B	H31B	120.00	.	.	.	no
C51B	C61B	H62B	120.00	.	.	.	no
C11B	C61B	H62B	120.00	.	.	.	no
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O1A	C1A	C8A	127.8(3)	.	.	.	no

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 C11B    C21B    C31B    C41B        0.0(4)     .       .       .       .   no
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C61A  H61A   O3A   0.93   2.48   3.080(3)   123   1_565 yes

#   END of Data for (2)

#------------------------------------------------------------------------------
# Reference: Smith & Wermuth (2012). J. Chem. Cryst.
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'SHELXL97 (Sheldrick, 2008) within WinGX (Farrugia, 1999)'
_computing_molecular_graphics     'PLATON (Spek, 2009)'
_computing_publication_material   'PLATON'
_refine_special_details
;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
;
```

```
_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0482P)^2^+0.1832P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     constr
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_abs_structure_details  ?

_refine_ls_abs_structure_Flack    ?
_refine_ls_number_reflns          1818
_refine_ls_number_parameters      222
_refine_ls_number_restraints      0
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_refine_ls_R_factor_gt            0.0433
_refine_ls_wR_factor_ref          0.1011
_refine_ls_wR_factor_gt           0.0971
_refine_ls_goodness_of_fit_ref    1.095
_refine_ls_restrained_S_all       1.095
_refine_ls_shift/su_max           0.002
_refine_ls_shift/su_mean          0.000
_refine_diff_density_max    0.175
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_atom_site_U_iso_or_equiv
_atom_site_calc_flag
_atom_site_refinement_flags
O1 O Uani 0.1037(3) 0.46332(13) 0.35912(11) 1.000 0.0367(6) . .
O3 O Uani 0.5369(5) 0.2897(2) 0.53034(15) 0.850 0.0476(9) . .
O31 O Uani 0.1366(3) 0.10171(14) 0.22248(11) 1.000 0.0380(6) . .
O32 O Uani 0.3926(3) 0.09139(14) 0.13149(11) 1.000 0.0390(6) . .
O41 O Uani 0.7330(3) 0.19428(16) 0.15843(12) 1.000 0.0426(6) . .
N2 N Uani 0.3471(3) 0.36865(14) 0.42859(12) 1.000 0.0261(6) . .
C1 C Uani 0.1777(4) 0.43430(17) 0.42344(16) 1.000 0.0296(7) . .
C3 C Uani 0.4017(6) 0.3497(3) 0.5113(2) 0.850 0.0307(10) . .
C4 C Uani 0.4201(5) 0.5129(3) 0.5883(2) 0.850 0.0428(11) . .
C5 C Uani 0.2979(6) 0.6088(3) 0.6144(2) 0.850 0.0466(11) . .
C6 C Uani 0.1545(6) 0.6428(3) 0.5474(2) 0.850 0.0474(11) . .
C7 C Uani -0.0030(8) 0.5595(4) 0.5263(3) 0.850 0.0413(11) . .
C8 C Uani 0.0946(5) 0.4534(2) 0.51132(18) 0.850 0.0305(9) . .
C9 C Uani 0.2771(5) 0.4215(3) 0.5648(2) 0.850 0.0311(9) . .
C11 C Uani 0.4455(3) 0.32000(18) 0.36025(14) 1.000 0.0259(6) . .
C21 C Uani 0.3407(4) 0.24678(17) 0.31485(13) 1.000 0.0248(6) . .
C31 C Uani 0.4341(3) 0.20192(17) 0.24630(14) 1.000 0.0259(6) . .
C41 C Uani 0.6356(4) 0.23185(19) 0.22519(15) 1.000 0.0296(7) . .
C51 C Uani 0.7424(4) 0.3035(2) 0.27390(16) 1.000 0.0339(8) . .
C61 C Uani 0.6485(4) 0.34691(18) 0.34041(16) 1.000 0.0315(7) . .
C311 C Uani 0.3206(4) 0.12700(18) 0.19463(15) 1.000 0.0281(7) . .
```

```
O3A O Uiso 0.601(2) 0.3292(11) 0.5195(10) 0.150 0.033(4) . .
C3A C Uiso 0.447(4) 0.3751(18) 0.4917(14) 0.150 0.033(6) . .
C4A C Uiso 0.319(3) 0.4723(13) 0.6274(11) 0.150 0.031(4) . .
C5A C Uiso 0.170(4) 0.5529(19) 0.6481(16) 0.150 0.062(6) . .
C6A C Uiso -0.028(3) 0.5489(18) 0.6050(13) 0.150 0.054(6) . .
C7A C Uiso 0.0360(10) 0.5520(7) 0.5151(6) 0.150 0.070(5) . .
C8A C Uiso 0.169(2) 0.4827(12) 0.4859(9) 0.150 0.017(3) . .
C9A C Uiso 0.316(4) 0.4464(19) 0.5354(15) 0.150 0.043(6) . .
H52 H Uiso 0.38830 0.66170 0.62950 0.850 0.0560 . .
H53 H Uiso 0.21320 0.58790 0.66230 0.850 0.0560 . .
H61 H Uiso 0.72010 0.39450 0.37250 1.000 0.0380 calc R
H62 H Uiso 0.08280 0.70600 0.56350 0.850 0.0570 . .
H63 H Uiso 0.23630 0.65890 0.49950 0.850 0.0570 . .
H71 H Uiso -0.09980 0.55290 0.57010 0.850 0.0490 . .
H72 H Uiso -0.07700 0.58180 0.47770 0.850 0.0490 . .
H43 H Uiso 0.50690 0.49050 0.63340 0.850 0.0510 . .
H8 H Uiso -0.01320 0.40100 0.52090 0.850 0.0370 . .
H9 H Uiso 0.22790 0.38400 0.61310 0.850 0.0370 . .
H21 H Uiso 0.20750 0.22700 0.32970 1.000 0.0300 calc R
H31 H Uiso 0.05640 0.05630 0.19590 1.000 0.0460 . R
H41 H Uiso 0.64500 0.14410 0.12700 1.000 0.0510 . .
H42 H Uiso 0.50840 0.53230 0.54360 0.850 0.0510 . .
H51 H Uiso 0.87780 0.32160 0.26100 1.000 0.0410 calc R
H8A H Uiso 0.25810 0.53830 0.46470 0.150 0.0210 . .
H9A H Uiso 0.40910 0.50460 0.52290 0.150 0.0520 . .
H41A H Uiso 0.28350 0.40980 0.65760 0.150 0.0380 . .
H42A H Uiso 0.45730 0.49320 0.64370 0.150 0.0380 . .
H51A H Uiso 0.23130 0.61980 0.63510 0.150 0.0750 . .
H52A H Uiso 0.14630 0.55120 0.70640 0.150 0.0750 . .
H61A H Uiso -0.12020 0.60440 0.62230 0.150 0.0650 . .
H62A H Uiso -0.09320 0.48250 0.61730 0.150 0.0650 . .
H71A H Uiso -0.09090 0.53840 0.48490 0.150 0.0840 . .
H72A H Uiso 0.07940 0.62280 0.50210 0.150 0.0840 . .

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O1 0.0288(9) 0.0385(10) 0.0429(10) 0.0071(9) -0.0094(8) 0.0051(8)
O3 0.0633(18) 0.0459(17) 0.0336(13) -0.0017(13) -0.0146(13) 0.0279(15)
O31 0.0301(9) 0.0446(11) 0.0393(10) -0.0141(9) -0.0023(8) -0.0126(9)
O32 0.0430(10) 0.0408(10) 0.0332(9) -0.0065(8) 0.0007(8) -0.0040(9)
O41 0.0322(9) 0.0501(11) 0.0454(11) -0.0075(10) 0.0071(8) -0.0004(9)
N2 0.0255(10) 0.0201(9) 0.0326(10) -0.0020(8) -0.0097(8) 0.0001(8)
C1 0.0251(12) 0.0192(11) 0.0445(14) -0.0040(11) -0.0114(11) -0.0030(9)
C3 0.042(2) 0.0246(17) 0.0255(17) -0.0001(14) -0.0051(15) 0.0024(15)
C4 0.0430(19) 0.0465(19) 0.0388(17) -0.0144(16) -0.0118(15) 0.0057(16)
C5 0.057(2) 0.0431(19) 0.0397(18) -0.0177(16) -0.0073(16) 0.0019(17)
C6 0.068(2) 0.0337(17) 0.0406(18) -0.0103(15) -0.0012(17) 0.0157(17)
C7 0.0288(19) 0.051(2) 0.044(2) -0.0127(18) -0.0032(15) 0.0185(16)
C8 0.0264(16) 0.0338(16) 0.0314(15) -0.0054(14) 0.0004(13) -0.0038(13)
C9 0.0392(17) 0.0316(17) 0.0226(15) 0.0002(14) 0.0000(13) 0.0066(14)
C11 0.0246(11) 0.0232(11) 0.0298(11) 0.0038(10) -0.0072(10) 0.0017(9)
C21 0.0227(11) 0.0250(11) 0.0266(11) 0.0044(9) -0.0041(9) -0.0029(10)
C31 0.0266(11) 0.0216(11) 0.0295(11) 0.0059(10) -0.0058(9) 0.0003(10)
C41 0.0264(12) 0.0304(12) 0.0320(12) 0.0033(10) -0.0006(10) 0.0031(10)
C51 0.0218(12) 0.0365(14) 0.0435(14) 0.0044(12) -0.0024(11) -0.0038(11)
```

```
C61 0.0282(12) 0.0248(12) 0.0415(14) 0.0011(11) -0.0098(11) -0.0032(10)
C311 0.0310(13) 0.0246(11) 0.0288(11) 0.0039(10) -0.0042(10) -0.0001(10)

_geom_special_details
;
 Bond distances, angles etc. have been calculated using the
 rounded fractional coordinates. All su's are estimated
 from the variances of the (full) variance-covariance matrix.
 The cell esds are taken into account in the estimation of
 distances, angles and torsion angles
;

loop_
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 O1      C1         1.218(3)                    .      .                 no
 O3      C3         1.206(5)                    .      .                 no
 O3A     C3A         1.24(3)                    .      .                 no
 O31     C311       1.315(3)                    .      .                 no
 O32     C311       1.225(3)                    .      .                 no
 O41     C41        1.353(3)                    .      .                 no
 O31     H31          0.8900                    .      .                 no
 O41     H41          1.0000                    .      .                 no
 N2      C3         1.424(4)                    .      .                 no
 N2      C1         1.384(3)                    .      .                 no
 N2      C11        1.433(3)                    .      .                 no
 N2      C3A         1.22(2)                    .      .                 no
 C1      C8         1.559(4)                    .      .                 no
 C1      C8A       1.200(15)                    .      .                 no
 C3      C9         1.506(5)                    .      .                 no
 C3A     C9A         1.44(3)                    .      .                 no
 C4      C5         1.523(5)                    .      .                 no
 C4      C9         1.542(5)                    .      .                 no
 C4A     C9A         1.55(3)                    .      .                 no
 C4A     C5A         1.45(3)                    .      .                 no
 C5      C6         1.503(5)                    .      .                 no
 C5A     C6A         1.46(3)                    .      .                 no
 C6      C7         1.516(6)                    .      .                 no
 C6A     C7A         1.53(2)                    .      .                 no
 C7      C8         1.519(6)                    .      .                 no
 C7A     C8A       1.326(16)                    .      .                 no
 C8      C9         1.527(5)                    .      .                 no
 C8A     C9A         1.33(3)                    .      .                 no
 C11     C61        1.395(3)                    .      .                 no
 C11     C21        1.377(3)                    .      .                 no
 C21     C31        1.401(3)                    .      .                 no
 C31     C41        1.401(3)                    .      .                 no
 C31     C311       1.477(3)                    .      .                 no
 C41     C51        1.400(4)                    .      .                 no
 C51     C61        1.368(4)                    .      .                 no
 C4      H43          0.9700                    .      .                 no
 C4      H42          0.9600                    .      .                 no
 C4A     H41A         0.9700                    .      .                 no
 C4A     H42A         0.9700                    .      .                 no
 C5      H52          0.9300                    .      .                 no
 C5      H53          1.0000                    .      .                 no
 C5A     H51A         0.9700                    .      .                 no
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```
 C5A     H52A          0.9700                    .       .                 no
 C6      H62           0.9700                    .       .                 no
 C6      H63           0.9700                    .       .                 no
 C6A     H61A          0.9700                    .       .                 no
 C6A     H62A          0.9700                    .       .                 no
 C7      H71           0.9600                    .       .                 no
 C7      H72           0.9700                    .       .                 no
 C7A     H71A          0.9700                    .       .                 no
 C7A     H72A          0.9700                    .       .                 no
 C8      H8            0.9800                    .       .                 no
 C8A     H8A           0.9800                    .       .                 no
 C9      H9            0.9800                    .       .                 no
 C9A     H9A           0.9800                    .       .                 no
 C21     H21           0.9300                    .       .                 no
 C51     H51           0.9300                    .       .                 no
 C61     H61           0.9300                    .       .                 no

loop_
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 C311    O31     H31          121.00              .       .       .          no
 C41     O41     H41          112.00              .       .       .          no
 C1      N2      C11         124.6(2)             .       .       .          no
 C1      N2      C3          111.0(2)             .       .       .          no
 C3A     N2      C11        117.3(12)             .       .       .          no
 C1      N2      C3A        115.4(11)             .       .       .          no
 C3      N2      C11         124.2(2)             .       .       .          no
 O1      C1      C8A         124.4(7)             .       .       .          no
 N2      C1      C8          108.2(2)             .       .       .          no
 N2      C1      C8A         107.4(7)             .       .       .          no
 O1      C1      N2          123.4(2)             .       .       .          no
 O1      C1      C8          128.3(2)             .       .       .          no
 N2      C3      C9          108.6(3)             .       .       .          no
 O3      C3      N2          122.4(3)             .       .       .          no
 O3      C3      C9          128.8(3)             .       .       .          no
 O3A     C3A     C9A           126(2)             .       .       .          no
 O3A     C3A     N2            135(2)             .       .       .          no
 N2      C3A     C9A         98.9(19)             .       .       .          no
 C5      C4      C9          111.9(3)             .       .       .          no
 C5A     C4A     C9A        111.9(18)             .       .       .          no
 C4      C5      C6          110.4(3)             .       .       .          no
 C4A     C5A     C6A           116(2)             .       .       .          no
 C5      C6      C7          112.2(3)             .       .       .          no
 C5A     C6A     C7A        103.2(16)             .       .       .          no
 C6      C7      C8          112.9(4)             .       .       .          no
 C6A     C7A     C8A        120.4(12)             .       .       .          no
 C7      C8      C9          117.9(3)             .       .       .          no
 C1      C8      C7          115.7(3)             .       .       .          no
 C1      C8      C9          103.0(2)             .       .       .          no
 C7A     C8A     C9A        118.4(16)             .       .       .          no
 C1      C8A     C9A        107.9(15)             .       .       .          no
 C4      C9      C8          113.8(3)             .       .       .          no
 C3      C9      C4          106.9(3)             .       .       .          no
 C3      C9      C8          104.0(3)             .       .       .          no
 C4A     C9A     C8A           122(2)             .       .       .          no
```

C3A	C9A	C4A	128(2)	.	.	.	no
C3A	C9A	C8A	110(2)	.	.	.	no
N2	C11	C21	120.16(19)	.	.	.	no
N2	C11	C61	119.5(2)	.	.	.	no
C21	C11	C61	120.3(2)	.	.	.	no
C11	C21	C31	120.2(2)	.	.	.	no
C21	C31	C41	119.1(2)	.	.	.	no
C21	C31	C311	121.0(2)	.	.	.	no
C41	C31	C311	119.9(2)	.	.	.	no
O41	C41	C31	122.4(2)	.	.	.	no
O41	C41	C51	117.8(2)	.	.	.	no
C31	C41	C51	119.8(2)	.	.	.	no
C41	C51	C61	120.3(2)	.	.	.	no
C11	C61	C51	120.2(2)	.	.	.	no
H43	C4	H42	108.00	.	.	.	no
C5	C4	H42	108.00	.	.	.	no
C9	C4	H43	108.00	.	.	.	no
C5	C4	H43	109.00	.	.	.	no
C9	C4	H42	111.00	.	.	.	no
H41A	C4A	H42A	108.00	.	.	.	no
C9A	C4A	H42A	110.00	.	.	.	no
C5A	C4A	H42A	110.00	.	.	.	no
C9A	C4A	H41A	109.00	.	.	.	no
C5A	C4A	H41A	108.00	.	.	.	no
C4	C5	H53	107.00	.	.	.	no
C6	C5	H53	108.00	.	.	.	no
H52	C5	H53	109.00	.	.	.	no
C6	C5	H52	112.00	.	.	.	no
C4	C5	H52	110.00	.	.	.	no
C4A	C5A	H51A	108.00	.	.	.	no
C4A	C5A	H52A	109.00	.	.	.	no
C6A	C5A	H52A	110.00	.	.	.	no
C6A	C5A	H51A	106.00	.	.	.	no
H51A	C5A	H52A	108.00	.	.	.	no
C7	C6	H62	109.00	.	.	.	no
H62	C6	H63	108.00	.	.	.	no
C5	C6	H63	109.00	.	.	.	no
C5	C6	H62	110.00	.	.	.	no
C7	C6	H63	109.00	.	.	.	no
H61A	C6A	H62A	108.00	.	.	.	no
C7A	C6A	H62A	110.00	.	.	.	no
C7A	C6A	H61A	115.00	.	.	.	no
C5A	C6A	H62A	108.00	.	.	.	no
C5A	C6A	H61A	112.00	.	.	.	no
C6	C7	H72	108.00	.	.	.	no
C6	C7	H71	109.00	.	.	.	no
C8	C7	H71	108.00	.	.	.	no
C8	C7	H72	110.00	.	.	.	no
H71	C7	H72	109.00	.	.	.	no
C6A	C7A	H71A	105.00	.	.	.	no
C8A	C7A	H72A	111.00	.	.	.	no
H71A	C7A	H72A	107.00	.	.	.	no
C8A	C7A	H71A	104.00	.	.	.	no
C6A	C7A	H72A	108.00	.	.	.	no
C1	C8	H8	107.00	.	.	.	no
C7	C8	H8	107.00	.	.	.	no
C9	C8	H8	106.00	.	.	.	no
C9A	C8A	H8A	93.00	.	.	.	no
C1	C8A	H8A	93.00	.	.	.	no
C7A	C8A	H8A	91.00	.	.	.	no
C8	C9	H9	110.00	.	.	.	no

```
 C3      C9      H9          110.00          .       .       .          no
 C4      C9      H9          111.00          .       .       .          no
 C8A     C9A     H9A          93.00          .       .       .          no
 C4A     C9A     H9A          92.00          .       .       .          no
 C3A     C9A     H9A          91.00          .       .       .          no
 C11     C21     H21         120.00          .       .       .          no
 C31     C21     H21         120.00          .       .       .          no
 C41     C51     H51         120.00          .       .       .          no
 C61     C51     H51         120.00          .       .       .          no
 C51     C61     H61         120.00          .       .       .          no
 C11     C61     H61         120.00          .       .       .          no
 O31     C311    O32        123.1(2)         .       .       .          no
 O31     C311    C31        114.2(2)         .       .       .          no
 O32     C311    C31        122.7(2)         .       .       .          no

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_geom_torsion_site_symmetry_3
_geom_torsion_site_symmetry_4
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 C3      N2      C1      O1          178.8(3)     .       .       .       .   no
 C3      N2      C1      C8           -6.1(3)     .       .       .       .   no
 C11     N2      C1      O1           -5.4(4)     .       .       .       .   no
 C11     N2      C1      C8          169.6(2)     .       .       .       .   no
 C1      N2      C3      O3          175.4(3)     .       .       .       .   no
 C1      N2      C3      C9           -9.0(3)     .       .       .       .   no
 C11     N2      C3      O3           -0.4(5)     .       .       .       .   no
 C11     N2      C3      C9          175.2(2)     .       .       .       .   no
 C1      N2      C11     C21         -65.7(3)     .       .       .       .   no
 C1      N2      C11     C61         114.7(3)     .       .       .       .   no
 C3      N2      C11     C21         109.6(3)     .       .       .       .   no
 C3      N2      C11     C61         -70.1(3)     .       .       .       .   no
 O1      C1      C8      C7          -37.0(4)     .       .       .       .   no
 O1      C1      C8      C9         -167.1(3)     .       .       .       .   no
 N2      C1      C8      C7          148.3(3)     .       .       .       .   no
 N2      C1      C8      C9           18.2(3)     .       .       .       .   no
 O3      C3      C9      C4           74.7(5)     .       .       .       .   no
 O3      C3      C9      C8         -164.6(4)     .       .       .       .   no
 N2      C3      C9      C4         -100.5(3)     .       .       .       .   no
 N2      C3      C9      C8           20.2(4)     .       .       .       .   no
 C9      C4      C5      C6          -57.9(4)     .       .       .       .   no
 C5      C4      C9      C3          158.6(3)     .       .       .       .   no
 C5      C4      C9      C8           44.5(4)     .       .       .       .   no
 C4      C5      C6      C7           61.4(4)     .       .       .       .   no
 C5      C6      C7      C8          -50.4(5)     .       .       .       .   no
 C6      C7      C8      C1          -84.6(4)     .       .       .       .   no
 C6      C7      C8      C9           37.8(5)     .       .       .       .   no
 C1      C8      C9      C3          -22.4(3)     .       .       .       .   no
 C1      C8      C9      C4           93.5(3)     .       .       .       .   no
 C7      C8      C9      C3         -151.1(3)     .       .       .       .   no
 C7      C8      C9      C4          -35.2(4)     .       .       .       .   no
 N2      C11     C21     C31         177.7(2)     .       .       .       .   no
 C61     C11     C21     C31          -2.7(3)     .       .       .       .   no
 N2      C11     C61     C51         -178.0(2)    .       .       .       .   no
 C21     C11     C61     C51            2.4(4)    .       .       .       .   no
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 C11     C21     C31     C41          0.6(3)     .       .       .       .   no
 C11     C21     C31     C311      -177.1(2)     .       .       .       .   no
 C21     C31     C41     O41       -177.9(2)     .       .       .       .   no
 C21     C31     C41     C51          1.8(3)     .       .       .       .   no
 C311    C31     C41     O41          -0.2(4)    .       .       .       .   no
 C311    C31     C41     C51         179.5(2)    .       .       .       .   no
 C21     C31     C311    O31          -5.0(3)    .       .       .       .   no
 C21     C31     C311    O32         175.2(2)    .       .       .       .   no
 C41     C31     C311    O31         177.3(2)    .       .       .       .   no
 C41     C31     C311    O32          -2.4(4)    .       .       .       .   no
 O41     C41     C51     C61         177.6(2)    .       .       .       .   no
 C31     C41     C51     C61          -2.1(4)    .       .       .       .   no
 C41     C51     C61     C11          0.1(4)     .       .       .       .   no

loop_
_geom_hbond_atom_site_label_D
_geom_hbond_atom_site_label_H
_geom_hbond_atom_site_label_A
_geom_hbond_distance_DH
_geom_hbond_distance_HA
_geom_hbond_distance_DA
_geom_hbond_angle_DHA
_geom_hbond_site_symmetry_A
_geom_hbond_publ_flag
#
#D   H   A   D - H  H...A   D...A    D - H...A  symm(A)
#
O31     H31     O1          0.8900     1.8200   2.712(3)     179.00   4_545 yes
O41     H41     O32         1.0000     1.7700   2.603(3)     138.00     .   yes
C21     H21     O31         0.9300     2.4300   2.739(3)     100.00     .   yes
C21     H21     O3          0.9300     2.5600   3.246(4)     131.00   3_456 yes
#  END of data for (3)
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